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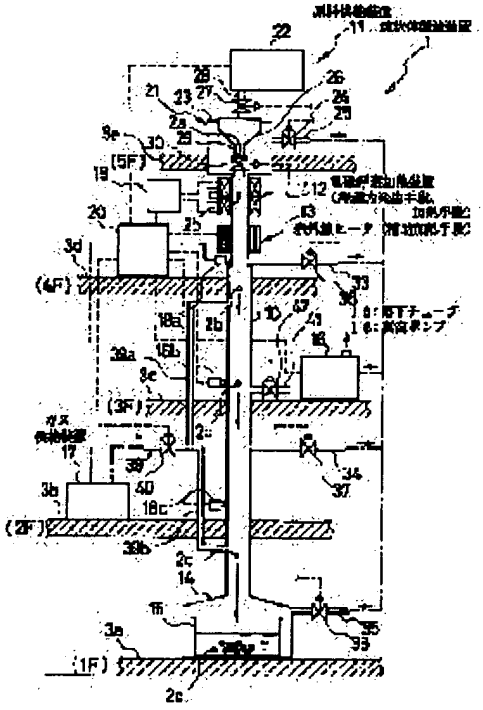
(54) MANUFACTURE OF SPHERICAL BODY OF INORGANIC MATERIAL AND MANUFACTURING APPARATUS THEREFOR

(57)Abstract:

PROBLEM TO BE SOLVED: To suppress the formation of projected parts in a part of the surface of a spherical body and lessen the inner strain in the spherical body by releasing heat, heating the surface part of a raw material molten liquid by an auxiliary heating means, and after that cooling the raw material at the time of dropping the raw material molten liquid in a dropping tube.

SOLUTION: When electricity is applied to an electromagnetic suspension heating apparatus 12 and a raw material body 2a is supplied to a dropping tube 10 while opening a shutter 30, the raw material body is suspended in floating state for a short time and heated and becomes a raw material molten liquid 2b. Next, electricity supply to the apparatus 12 is stopped, the raw material molten liquid 2b starts dropping in the tube 10 and is cooled by radiation cooling until is reaches the upper end level of an infrared heater 13 and releases heat. At that time, the raw material molten liquid 2b becomes truly spherical state. After that, only the surface of the raw material molten liquid 2b of the heater 13 is heated and the raw material molten liquid 2b is solidified to be a spherical crystal 2c with a truly spherical shape. Finally, the spherical crystal 2c drops to silicon oil in a silicon oil tank 15 and is completely cooled. In this way, a spherical body in which components are uniformly distributed can be manufactured.



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* NOTICES *

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2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] Especially this invention relates to the technique of manufacturing the spherule made from an inorganic material economically comparatively simply, about the technique of making a spherule solidifying the raw material object of inorganic materials, such as a semi-conductor, a superconductor, the magnetic substance, a dielectric, an alloy, and glass, in an operation of surface tension, carrying out free fall of the inside of an after [melting] fall tube in the state of suspension, about the manufacture approach of the spherule made from an inorganic material, and its manufacturing installation.
[0002]
[Description of the Prior Art] Since melting of the inorganic materials various in the bottom of a minute gravity environment is carried out conventionally, the experiment which a spherule is made to solidify has been conducted. The experiment which makes a spherical crystal was also conducted into it. as the melting approach of not using a container for the bottom of a minute gravity environment -- electromagnetism -- the technique which combined synchrotron orbital radiation heating furnaces, such as a halogen lamp, with suspension heating, electrostatic suspension, or an acoustic wave swimming device has been used. However, since such an experiment was conducted under the minute gravity environment of long duration using the satellite and space shuttle on [instead of the ground] an orbit around the earth, great costs and time amount are taken, and also the constraint on operation is also severe and the count of an experiment is limited. Therefore, it is not suitable for it being restricted to scientific research or an experiment, repeating the spherical crystal of an above-mentioned inorganic material economically for a short time, and producing in large quantities.
[0003] On the other hand, the minute gravity environment of a short time for 10 or less seconds of it being realizable with the drop tube prepared in the conventional ground and the experimental device of a shot tower method is well-known. for example, in the drop tube type experimental device of NASA of the U.S., a drop tube equips on the ground -- having -- the bell jar of the upper limit section -- electromagnetism -- the ** which suspension heating apparatus is equipped exchangeable and does not pay the sample of various inorganic materials to a container -- electromagnetism -- suspension heating is carried out, and a spherule is made to fuse, and to solidify under minute gravity, carrying out free fall of the inside of the vacuum of a drop tube
[0004] Moreover, the example which made the spherical crystal of silicon using the shot tower is announced ["Development and Evaluation of the Texas Instruments Solar Energy System" 16 th IEEE PVSC Proceedings P.257 -P.260 (1982)]. According to this announcement paper, from the small nozzle with which the upper limit of a shot tower was equipped, the melt of silicon is injected small quantity every, free fall of the inside of the inert gas in a shot tower is carried out, and the spherical crystal of silicon is manufactured. The technique of injecting the melt of silicon small quantity every, carrying out free fall of the inside of a shot tower, and manufacturing the spherical crystal of silicon from the small nozzle with which the U.S. Pat. No. 4,021,323 official report as well as the above was equipped at the

upper limit of a shot tower is indicated.
[0005]
[Problem(s) to be Solved by the Invention] As mentioned above, in making the melt of silicon inject from a nozzle and manufacturing the spherical crystal of silicon, it is not suitable for possibility that an impurity will melt into silicon melt from a nozzle being high, and manufacturing the silicon spherical crystal of a high grade. This is also the same as when manufacturing the spherical crystals and spherules made from an inorganic material other than silicon. however, the drop tube type experimental device of NASA -- like -- electromagnetism -- if suspension heating apparatus is applied, since silicon melt will not be contacted in a container, an impurity does not melt into silicon melt
[0006] On the other hand, when carrying out free fall of the inside of a vacuum or inert gas and making the melt of silicon solidify, in order to radiate heat from the front face of silicon melt, coagulation begins from the front-face side of silicon melt. However, since a part of silicon melt bulges in the piece place by the side of a front face and a tail-like height is formed when the interior of melt solidifies later than a front-face side, in order to carry out cubical expansion at the time of coagulation, silicon cannot form a real ball-like silicon spherical crystal. A crevice may be formed in the surface section of the spherule which was solidified contrary to the above in the case of the inorganic material which carries out a volumetric shrinkage at the time of coagulation. Moreover, if the front-face side of silicon melt solidifies previously, it will become easy to mix the air bubbles adhering to a silicon raw material object in the interior of a spherule.
[0007] And since silicon melt congeals from a front-face side, the internal distortion of the spherical crystal after coagulation also becomes large. When manufacturing the spherical crystal of silicon, annealing processing can remove said internal distortion separately. However, in the case of the inorganic material which cannot carry out the formation postheat treatment of the spherule, it is difficult to remove the internal distortion of a spherule. Anyway, in order to raise the quality of a spherical crystal or a spherule, it is desirable to make internal distortion small as much as possible from the formation phase of a spherical crystal or a spherule.
[0008] In case the purpose of this invention manufactures the spherule made from an inorganic material, it is offering the manufacture approach of the spherule which can control a tail-like height being formed in a part of front face of a spherule, can make internal distortion of a spherule small, and can prevent mixing of the air bubbles inside a spherule, and its manufacturing installation.
[0009]
[Means for Solving the Problem] The manufacture approach of the spherule made from the inorganic material of claim 1 The 1st process to which the raw material object which consists of an inorganic material is heated with a heating means, and carries out melting in the condition of having made it floating in a vacuum or predetermined gas with a suspension force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the fall tube which made raw material melt the vertical posture, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said fall tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said fall tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, it is characterized by having the 5th process which holds said spherule in the coolant of the cooling cistern which attends the lower limit of said fall tube.
[0010] as said suspension force generating means -- electromagnetism -- suspension heating apparatus, an electrostatic swimming device, an acoustic wave swimming device, etc. -- applicable -- as said heating means -- electromagnetism -- suspension heating apparatus, an infrared heater, an electric heater, a laser beam heater, a halogen lamp, etc. are applicable. As predetermined gas, oxidizing gases, such as nitrogen gas containing inert gas, such as argon gas, gaseous helium, and nitrogen gas, oxygen gas, or oxygen gas, are applicable. Inorganic materials are which ingredients, such as a semi-conductor, a superconductor, the magnetic substance, a dielectric, an alloy, and glass.
[0011] At the first process [1st], the minute massive raw material object of an inorganic material is

supplied to a suspension force generating means by a parts feeder etc., by the condition of having made the raw material object floating in a vacuum or predetermined gas with the suspension force generating means, it heats with a heating means and melting is carried out. Thus, in order to carry out and change melting of the raw material object into a suspension condition, in order that raw material melt may not contact a container, an impurity does not melt into raw material melt. Heat is made to radiate at the 2nd following process with a melt condition, dropping the inside of the vacuum in the fall tube which made raw material melt the vertical posture, or predetermined gas.

[0012] In order to carry out free fall of the inside of a fall tube for raw material melt, raw material melt goes into a minute gravity condition, it becomes spherical in an operation of the surface tension of raw material melt, and in order that there may be no effect of gravity or a heat convection, a component serves as melt distributed over homogeneity. When dropping the inside of a vacuum, heat is radiated from the front face of raw material melt by radiation, and when dropping the inside of predetermined gas, heat is radiated from the front face of raw material melt with radiation and heat transfer. By making extent which extent which the coagulation of the front face of said raw material melt does not generate, or partial coagulation does not generate radiate heat, the temperature fall of the whole raw material melt is aimed at. However, in order to radiate heat from the front-face side of raw material melt, the direction of the surface section becomes low temperature from the interior of raw material melt.

[0013] At the 3rd following process, while raw material melt drops the inside of the vacuum in a fall tube, or predetermined gas, the surface section of raw material melt is heated with an auxiliary heating means. As this auxiliary heating means, in order to heat only the surface section, it is desirable to apply an infrared heater, and it heats so that the temperature of the surface section may become high rather than the temperature inside raw material melt.

[0014] It cools dropping the inside of the vacuum in a fall tube, or predetermined gas, and a spherule is made to solidify raw material melt in an operation of the surface tension of semi-conductor melt in the 4th following process. It solidifies to a spherule, holding the shape of a ball in an operation of surface tension, in order that raw material melt may congeal carrying out free fall. Although raw material melt radiates heat from the surface section at this time, since the temperature inside raw material melt is lower on a par with the temperature of the surface section than the temperature of the surface section, coagulation begins from both the interior and the surface section from the interior of raw material melt. So, also in the case of the inorganic material which can control effectively that a height is formed in a part of front face of a spherule, and carries out a volumetric shrinkage at the time of coagulation, it can control effectively that a crevice is formed in a part of front face of a spherule, and, also in the case of the inorganic material which carries out cubical expansion at the time of coagulation, internal distortion of a spherule can be made small. Moreover, it is hard coming to mix air bubbles in the spherical inside of the body. And in order to solidify from the condition that there is no seed crystal used as the origin of coagulation initiation, the coagulation in a supercooling condition arises. When an inorganic material is glass, it becomes the spherule of completely new glass by supercooling coagulation.

[0015] Although a part of melt will project in the surface section and a height will be formed in case the interior of raw material melt solidifies if it begins to solidify from the surface section of raw material melt in order to carry out cubical expansion of the silicon especially when an inorganic material is silicon, and solidifying In this invention, even if such a height is not formed and a height is formed, a ********** height to the extent that it disappears separately in the case of annealing processing will only be formed.

[0016] At the 5th following process, a spherule is held in the coolant of the cooling cistern which attends the lower limit of a fall tube. As this coolant, the liquid (for example, silicone oil) which does not make an impurity melt into a spherule is applied. Thus, by holding the spherule which has fallen in the coolant, a buffer can be aimed at and a spherule can fully be cooled.

[0017] The manufacture approach of the spherule made from the inorganic material of claim 2 is characterized by said inorganic material being silicon in invention of claim 1. It can control that a projection is formed in the surface section of a silicon spherical crystal, and internal distortion of a spherical crystal can be made small as explained in the column of said claim 1.

[0018] The manufacture approach of the spherule made from the inorganic material of claim 3 The 1st process to which two or more raw material objects which consist of an inorganic material of a mutually different class are heated with a heating means, and carry out melting in one in the condition of having made it floating in the shape of contact in a vacuum or predetermined gas with a suspension force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the fall tube which made raw material melt the vertical posture, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said fall tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said fall tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, it is characterized by having the 5th process which holds said spherule in the coolant of the cooling cistern which attends the lower limit of said fall tube.
[0019] Although this manufacture approach is fundamentally [as the manufacture approach of claim 1] the same fundamentally, in the 1st process, it differs at the point which applies two or more raw material objects which consist of an inorganic material of a class which is mutually different, and is the same as that of claim 1 about the 2nd process - the 5th process. That is, in the condition of having made it floating in the shape of contact in a vacuum or predetermined gas with a suspension force generating means, two or more raw material objects which consist of an inorganic material of a class which is mutually different in the 1st process are heated with a heating means, and carry out melting in one. The weight ratio of two or more raw material objects does not restrict that it is the same, but is set up suitably. Since it is the same as that of claim 1 about said inorganic material, a suspension force generating means, and a heating means, explanation is omitted. According to this manufacture approach, it is the spherule which consists of an inorganic material with which two or more classes differ, the spherule from which the component was distributed over homogeneity can be manufactured, it can control that a height is formed in the surface section of a spherule, and internal distortion of a spherule can be made small.
[0020] The manufacture approach of the spherical crystal made from the inorganic material of claim 4 is characterized by the inorganic materials of a class which is different in mutual [said] being silicon and germanium in invention of claim 3. Since silicon and germanium form a complete solid solution, the spherical crystal of the silicon germanium mixed-crystal semiconductor which has a desired mixed-crystal ratio can be manufactured by choosing a presentation ratio as arbitration.
[0021] The manufacturing installation of the spherule made from the inorganic material of claim 5 In the equipment which heats the raw material object which consists of an inorganic material in the state of suspension in the vacuum in a fall tube, or predetermined gas, is made to solidify the raw material melt, carrying out free fall of the inside of a fall tube, and manufactures a spherule The fall tube of a vertical posture, and a raw material object supply means to supply a raw material object from the upper limit into said fall tube, The suspension heating means which heats in the condition of having made the raw material object floating in the upper limit section of said fall tube, or its neighborhood, and is made into raw material melt, It is isolated to said suspension heating means down side beyond predetermined distance, is arranged in it, and has the after heater which heats the surface section of the raw material melt under fall [inside / of a fall tube].
[0022] Said inorganic materials are any one ingredient or two or more ingredients, such as a semiconductor, a superconductor, the magnetic substance, an alloy, and glass, and the raw material object of one kind of ingredient, the raw material object of two or more ingredients, one raw material object, two or more raw material objects, etc. can apply the raw material object of a gestalt variously as a raw material object. About predetermined gas, it is the same as that of claim 1. as said suspension heating means -- electromagnetism -- it can apply any of suspension heating apparatus, an electrostatic swimming device, heating apparatus and an acoustic wave swimming device, and heating apparatus ** they are, and various heating means, such as an electric heater, an infrared heater, a halogen lamp heater, and a laser beam heater, can be applied as said heating apparatus. As said after heater, various heaters, such as an infrared heater, a halogen lamp heater, a laser beam heater, and an electric heater, are

applicable.
[0023] If a feeding means supplies a raw material object from the upper limit into a fall tube, a suspension heating means will be heated in the condition of having made the raw material object floating in the upper limit section of a fall tube, or its neighborhood, and will be made into raw material melt. Since raw material melt is in a suspension condition, and a container is not contacted, an impurity does not melt into raw material melt. Although raw material melt radiates heat by radiative cooling etc. and the temperature of the whole raw material melt falls while raw material melt falls the inside of a fall tube to the level of an after heater since an after heater is isolated to the suspension heating means down side beyond predetermined distance and is arranged in it, the direction of the surface section becomes low temperature from the interior of raw material melt.
[0024] Next, since the surface section of the raw material melt is heated at an after heater, the direction of the surface section becomes an elevated temperature from the interior of raw material melt. Since the temperature inside raw material melt is lower on a par with the temperature of the surface section than the temperature of the surface section when a temperature fall is carried out to the congealing point, in order to radiate heat from the surface section of raw material melt during fall of raw material melt, after the raw material melt passes an after heater, coagulation begins from both the interior and the surface section from the interior of raw material melt. Consequently, the operation explained to claim 1 and the same operation are done so.
[0025] The manufacturing installation of the spherule made from the inorganic material of claim 6 establishes the vacuation means which makes the inside of said fall tube a vacua through a vacuum pump in invention of claim 5. In case the inside of a fall tube is made into a vacua and a spherule is manufactured, a vacuation means is operated and the inside of a fall tube is made into a vacua.
[0026] In invention of claim 6, the manufacturing installation of the spherule made from the inorganic material of claim 7 establishes a gas supply means to form the gas stream which flows by this ** mostly in the fall direction of raw material melt in a fall tube, and to form the gas stream which flows to the fall direction and opposite direction of a spherule in a fall tube while supplying the predetermined gas according to the class of inorganic material in a fall tube. For example, when manufacturing a glass spherule and the spherule made from an oxide high-temperature superconductor, a vacuation means will be stopped, the nitrogen gas containing oxidization gas or oxygen gas will be supplied in a fall tube, and a spherule will be manufactured in the gas. In that case, the gas stream which forms the gas stream which flows by this ** mostly in the fall direction of raw material melt in a fall tube, and flows to the fall direction and opposite direction of a spherule in a fall tube with a gas supply means while supplying gas in a fall tube is formed.
[0027] Although the raw material melt of an about several 100-2000-micrometer minor diameter is solidified in a **** short time within a fall tube, in order to form the gas stream which flows by this ** mostly in the fall direction of the raw material melt before coagulation, frictional force hardly acts on the raw material melt under coagulation from a gas stream, but raw material melt is solidified in the shape of a real ball. And in order to form the gas stream which flows to the fall direction and opposite direction of a spherule after coagulation, the contact degree of a gas stream and a spherule becomes high, and the reaction of gas and a spherule and cooling of a spherule are promoted.
[0028] In invention of claim 7, the lower limit of said fall tube is faced the manufacturing installation of the spherule made from the inorganic material of claim 8, and it forms the cooling cistern which holds in the coolant the spherule which fell out of the lower limit. A spherule can be cooled while preventing that a spherule is damaged with an impact, in order to hold the spherule which fell out of the lower limit of a fall tube in the coolant of a cooling cistern.
[0029]
[Embodiment of the Invention] Hereafter, it explains, referring to a drawing about the gestalt of operation of this invention. The spherule manufacturing installation which first manufactures the spherule made from an inorganic material (the number of diameters 100-2000 micrometers) applied to this invention is explained. As shown in drawing 1 , the spherule manufacturing installation 1 for the diameter of 5-10cm The fall tube 10 of a vertical with a height of about 14m, the electromagnetism

arranged on the outside of the upper limit section of the fall tube 10 -- with the suspension heating apparatus 12 The infrared heater 13 (auxiliary heating means) as an after heater, and the feeding equipment 11 which supplies one raw material object 2a at a time to the upper limit of the fall tube 10, The silicone oil tub 15 which holds in the hold section 14 which stands in a row in the lower limit of the fall tube 10, and attends the lower limit of the fall tube 10, The vacuum pump 16 which attracts the air in the fall tube 10, and a gas transfer unit 17, It consists of high speed cameras 18a-18c which photo the pipe line and bulbs, raw material melt 2b under fall [inside / of the fall tube 10], and spherical crystal 2c (spherule), and control unit 20 grade which controls these devices. In addition, the floor [first] - the floor [fifth] floors 3a-3e of works are also illustrated.

[0030] Feeding equipment 11 is equipped with the parts feeder 22 which holds raw material object 2a of the shape of a pellet of the feeder 21 connected to the upper limit of the fall tube 10, and much predetermined sizes, and it supplies at a time to one feeder 21, and the parts feeder 22 is constituted so that degasification of the air of the front face of raw material object 2a may be carried out, while heating raw material object 2a beforehand. the case 23 of a feeder 21 -- electromagnetism -- it connects with a vacuum pump 16 by the siphon 25 which has the closing motion valve 24 -- having -- the acceptance machine 26 within a case 23 -- electromagnetism -- it connects with a parts feeder 22 at the path 28 which has the closing motion shutter 27 -- having -- the outlet path 29 of the acceptance machine 26 -- electromagnetism -- the closing motion shutter 30 is formed and the vacuum within a case 23 is introduced into the acceptance machine 26 through two or more micropores.

[0031] under operation of the spherule manufacturing installation 1 -- electromagnetism -- the closing motion valve 24 is opened and is a vacua in a feeder 21. the case where one raw material object 2a is supplied to a feeder 21 from a parts feeder 22 -- electromagnetism -- the closing motion shutter 30 -- closing -- electromagnetism -- the closing motion shutter 27 -- opening -- the inside of the acceptance machine 26 -- raw material object 2a -- supplying -- after that -- electromagnetism -- the closing motion shutter 27 is closed. Supply of feeding equipment 11 is attained to the feeder 21 in one raw material object 2a at every predetermined time (for example, 1 second).

[0032] the siphon 33-35 which attracts the air in the fall tube 10 -- electromagnetism -- the closing motion valves 36-38 are equipped, and these siphon 33-35 is connected to the vacuum pump 16. So that predetermined gas (inert gas, oxidizing gas, etc.) can be passed in the fall tube 10 depending on the class of inorganic material The gas supply line 39 prolonged from a gas transfer unit 17 and its gas transfer unit 17 is formed.